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ENVIRONMENTAL PROTECTION AGENCY

40 CFR Part 136

[EPA-HQ-OW-2010-0192; FRL-9787-7]

Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Analysis and Sampling Procedures; Notice

AGENCY: Environmental Protection Agency (EPA).

ACTION: Notice of final decision.

SUMMARY: EPA discussed, but did not propose, a new method, ASTM D7575, for oil and grease in the 2010 proposed Methods Update Rule (MUR). Oil and grease is a method-defined parameter. That is, the nature and amount of material determined by the method is defined in terms of the method. EPA subsequently published a Notice of Data Availability (NODA) on this method that provided new data and requested comment on whether and how EPA should approve the method in Part 136 as an alternative oil and grease method. This document provides EPA's final decision on its reconsideration of this method.

DATES: [insert date of publication in the Federal Register].

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SUPPLEMENTARY INFORMATION:

I. Background

A. CWA Analytical Methods and Limited Use Alternate Test Procedures (ATP)

Program

EPA establishes test procedures (also referred to as analytical methods) codified in 40 CFR Part 136 under its authority in section 304(h) of the CWA to promulgate guidelines establishing test procedures for the analysis of pollutants. EPA's regulations provide that, when EPA has promulgated a test procedure for analysis of a specific pollutant in 40 CFR Part 136, an NPDES permittee must use an approved test procedure for the specific pollutant when measuring the pollutant for an application submitted to EPA or to a State with an approved NPDES program and for reports required to be submitted by dischargers under the NPDES program. See 40 CFR § 136.1(a). This approach simplifies the permitting process for hundreds of thousands of NPDES and indirect discharging permittees and permitting authorities. In the absence of an approved test procedure for a specific pollutant (or when an approved test procedure does not work in a specific matrix, e.g., because of a matrix interference), generally, a permit applicant may use any suitable method but must provide the permitting authority a description of the method for evaluation of its suitability. See 40 CFR 122.21(g)(7). However, 40 CFR Part 136 also recognizes that new technologies and approaches are constantly being developed, including methods for pollutants for which EPA already has an approved test procedure. As such, Part 136.5 allows for use of an alternate method for a specific pollutant or parameter in a regulated CWA matrix that is different from the approved test procedure (i.e., limited use approval). Requests for such uses, along with supporting data, are made to the applicable Regional Alternate Test Procedure (ATP) Coordinator for consideration and approval.

B. Oil and Grease

Unlike many parameters, oil and grease is not a unique chemical entity, but is a mixture

of chemical species that varies from source to source. Common substances that may contribute to oil and grease include petroleum based compounds such as fuels, motor oil, lubricating oil, soaps, waxes, and hydraulic oil and vegetable based compounds such as cooking oil and other fats. Oil and grease is defined by the method used to measure it (i.e., it is a method-defined analyte). The CWA defines oil and grease as a conventional parameter and hundreds of thousands of NPDES permits and indirect discharging permits contain oil and grease numerical limits. Currently, Part 136 lists two analytical methodologies for the measurement of oil and grease in such discharge permits. Permittees have been using EPA Method 1664A to measure compliance with such discharge limits. Method 1664A is a liquid/liquid extraction (LLE), gravimetric procedure that employs normal hexane (*n*-hexane) as the extraction solvent that is applicable for measuring oil and grease in concentrations from 5 mg/L to 1,000 mg/L. This method also allows the use of solid-phase extraction (SPE) provided that the results obtained by SPE are equivalent to the results obtained by LLE.

C. Method-defined Analytes

The measurement results obtained for a method-defined analyte are both specific to that method and solely dependent on the method used. As a consequence, the results obtained for a parameter defined by one particular method are not necessarily directly comparable to results obtained by another method (i.e., the data derived from method-defined protocols cannot be reliably verified outside the method itself). EPA has defined a method-defined analyte in 40 CFR 136.6(a)(5) as "...an analyte defined solely by the method used to determine the analyte. Such an analyte may be a physical parameter, a parameter that is not a specific chemical, or a parameter that may be comprised of a number of substances. Examples of such analytes include temperature, oil and grease, total suspended solids, total phenolics, turbidity, chemical oxygen

demand, and biochemical oxygen demand.”

D. EPA’s Past Consideration of Alternative Oil and Grease Methods for Adoption in 40 CFR Part 136

Because oil and grease is a method-defined parameter, EPA has not considered promulgating multiple methods to measure oil and grease that are based on different extractants. Moreover, EPA has not considered multiple oil and grease methods that are based on different determinative techniques. The only exception to this was EPA’s promulgation of EPA Method 1664A in 1999 to replace EPA Method 413.1 (64 FR 26315), a similar procedure that used Freon[®] (1,1,2-trichloro-1,2,2-trifluoroethane (CFC-113; Freon-113)) as the extraction solvent. EPA made this exception because Freon[®] was banned by an international treaty (the Montreal Protocol on Substances That Deplete the Ozone Layer), and until the ban went into effect, EPA allowed either of these oil and grease methods for CWA compliance. In both methods, the determinative technique is gravimetry and the only change was the extraction solvent (n-hexane instead of Freon[®]).

When EPA promulgated EPA Method 1664A to replace EPA Method 413.1, EPA evaluated a variety of possible replacement extracting solvents in addition to n-hexane. EPA selected n-hexane and promulgated Method 1664A after conducting multi-year, extensive side-by-side studies on a variety of samples representing a wide range of matrices/discharges (see “Preliminary Report of EPA Efforts to Replace Freon for the Determination of Oil and Grease,” EPA-821-R-93-011, September 1993, and "Report of EPA Efforts to Replace Freon for the Determination of Oil and Grease and Total Petroleum Hydrocarbons: Phase II," EPA-820-R-95-003, April 1995). In considering which solvent produced results most comparable to results obtained with Freon[®], EPA conducted a Root Mean Squared Deviation (RMSD) evaluation of

the data collected in the side-by-side studies. None of the alternative solvents produced results statistically comparable to results produced by Freon[®]. However, EPA concluded at the time that n-hexane was appropriate as an alternative solvent, based on overall extraction results (96% versus 100% for Freon) and analytical practical considerations (e.g., boiling point).

To accommodate concerns about possible differences in results, EPA allowed permitting authorities to establish a conversion factor by having the discharger perform a side-by-side comparison of Method 1664A and the Freon[®] extraction method and then adjusting the discharge limits, if necessary, to account for differences in the permit. EPA further recommended a specific process to follow for the side-by-side comparison in the guidance document for Method 1664A (see "Analytical Method Guidance for EPA Method 1664A Implementation and Use (40 CFR part 136)," EPA/821-R-00-003, February 2000).

E. Proposed 2010 Methods Update Rule (MUR)

On September 23, 2010, EPA proposed to add new and revised EPA methods to its Part 136 test procedures (75 FR 58024). Among other methods, in the September 2010 proposal, EPA described three oil and grease methods published by ASTM International or the Standard Methods Committee that require a different extractant and/or a different measurement (i.e., determinative) technique than the existing Part 136 oil and grease methods. These methods were ASTM D7575, ASTM D7066 and Standard Methods 5520. Section 12(d) of the National Technology Transfer and Advancement Act (NTTAA) of 1995 (Public Law 104-113; 15 U.S.C. 272 note) directs EPA to use voluntary consensus standards in its regulatory activities unless to do so would be inconsistent with applicable law or is otherwise impractical. As such, when requested by ASTM and Standard Methods to include their methods in 40 CFR Part 136, EPA may propose to approve a method or explain why it should or should not do so. The proposal

explained the issues surrounding method-defined parameters, and explained that, consistent with past practices, EPA was not proposing to include any of the three oil and grease methods in Part 136, including ASTM D7575.

F. December 2011 Notice of Data Availability (NODA)

In response to the September 2010 proposal, EPA received comments recommending that it reconsider alternative methods for oil and grease. Some of the comments focused exclusively on the oil and grease method ASTM D7575. Unlike EPA Method 1664A which uses n-hexane as the extractant and gravimetry for the measurement of the extracted materials, ASTM D7575 uses an extracting membrane followed by infrared measurement of the sample materials that can be retained on the membrane. In particular, commenters cited that ASTM D7575 is solvent free and provides reliable and comparable results to EPA Method 1664A. These commenters submitted additional information on the health hazards associated with hexane as well as additional single laboratory comparability data between Method 1664A and ASTM D7575 and on additional matrices tested after the initial comparability study and associated statistical analysis.

Because EPA is interested in promoting the use of solvent-free methods and this new information, EPA re-evaluated the ASTM D7575 method for the measurement of oil and grease and published a Notice of Data Availability on December 14, 2011 (76 FR 77742). The notice provided the additional data and EPA's analysis of that data. Further, it explained that, after evaluating the new information, EPA was re-considering its decision not to include ASTM D7575 in 40 CFR Part 136 as an alternative to EPA Method 1664A for measuring oil and grease. The notice explained that EPA had three primary reasons for this re-consideration. First, EPA's analysis demonstrates ASTM D7575 is an acceptable stand alone method for the measurement of oil and grease in wastewater for its applicable reporting range (5-200 mg/L). Second, it produces

results that, while not statistically comparable across all matrices tested¹, are generally very close to those obtained using EPA Method 1664A for the matrices tested. Third, EPA supports pollution prevention, and is particularly persuaded by the substantial advantages associated with the green aspects of this membrane technology (e.g., it uses a solventless extraction, there is no solvent waste, and no exposure of the analyst to solvent).

However, the notice also discussed implementation considerations associated with promulgating an alternative method based on a different determinative step for a method-defined parameter. EPA recognized the potential impact that this new method could have on the hundreds of thousands of oil and grease determinations in regulatory Clean Water Act programs, and, as such, was keenly interested in obtaining additional input from stakeholders. Therefore, the notice explained that, while EPA determined that the results of the EPA 1664A and ASTM D7575 are comparable over the applicable range where the two methods overlap (5-200 mg/L) for the set of the 13 wastewater matrices evaluated, it continued to have compliance concerns with promulgating the ASTM method for nationwide use as an alternative to EPA Method 1664A. More specifically, because oil and grease measures a wide variety and type of individual compounds and because oil and grease is extensively incorporated in permits covering a wide variety of wastewater matrices, the result of promulgating the ASTM D7575 method as an alternative to EPA Method 1664A is that a permittee could be in or out of compliance simply due to a change in the test method used to evaluate samples.

Finally, through the notice, EPA requested comment on its conclusion that ASTM D7575 is an acceptable choice for the determination of oil and grease, and whether it should reconsider its policy towards method-defined parameters for this particular method. In particular, the notice

¹ Similar to the approach it used when it replaced Freon with hexane, EPA performed a RMSD evaluation of the ASTM D-7575 results and EPA Method 1664A results for the available matrices evaluated. See 76 FR 77745.

requested comment on whether or not EPA should reconsider promulgating this specific additional method for oil and grease based on different extractants and determinative techniques than EPA Method 1664A. Further, in the event that EPA were to promulgate this specific alternative method, the notice requested comment on some approaches that could ensure comparability for individual permittees (e.g., EPA requested comment on the need for a permit adjustment based on side-by-side comparison of Methods 1664A and ASTM D7575).

G. Summary of NODA Comments

EPA received comments from 106 stakeholders. Approximately, a third expressed support for nationwide approval of the ASTM D7575 method as an alternative oil and grease method. In general, these comments were similar to those received on EPA's proposal: ASTM D7575 is "green" (e.g., less hazardous waste, no exposure to toxic chemicals), it is easier, faster, less expensive and potentially portable in comparison to EPA 1664A, and it produces results substantially in agreement with Method 1664A. About two-thirds of the comments recommended EPA not approve it for use as an alternative oil and grease method. Some comments were specific to the sampling requirements and sample preparation procedures of the method, raising technical concerns such as the representativeness of the 10 mL aliquot and concerns over the drying procedures. Some comments were more overarching such as comments that ASTM D7575 had not been tested in a sufficient number of matrices. Others were specific to the consideration of the ASTM D7575 method as an alternative to EPA method 1664A, such as the applicable range of the ASTM D7575 method (5 to 200 mg/L) was limited in comparison to EPA Method 1664A. Some noted that the ASTM method did not produce statistically comparable results to EPA Method 1664A and EPA should retain its policy not to approve alternative methods for method-defined parameters that are not based on the same determinative

step. Finally, many shared the concerns raised in the notice about implementing ASTM D7575 on a nationwide basis as an alternative oil and grease method and the possibility that a discharger could be in or out of compliance simply by changing the method.

III. Final Determination on ASTM D7575 as an Alternative to Existing Part 136 Oil and Grease Methods

As explained in the NODA, EPA's consideration of ASTM D7575 represents a unique situation. Because oil and grease is a method-defined parameter, EPA has not considered promulgating multiple methods to measure oil and grease that are based on different extractants. Moreover, EPA has not considered multiple oil and grease methods that are based on different determinative techniques. The only exception to this was EPA's promulgation of EPA Method 1664A to replace Method 413.1, a similar procedure that used (the internationally banned extraction solvent) Freon[®]. Thus, EPA's consideration of ASTM D7575 as an alternative oil and grease method represents a new path for EPA. As is always the case, EPA proceeded carefully, with a particular focus on the underlying data. This consideration is specific to ASTM D7575 and should not be interpreted broadly to other oil and grease methods that use different extractants and/or determinative techniques, or more generally to other method-defined analytes. If EPA receives similar requests for other methods, it will evaluate each one individually.

A. ASTM D7575 is a Good Stand Alone Method for the Measurement of Oil and Grease in Aqueous Matrices

After careful consideration of all the comments received on the NODA, EPA continues to conclude that ASTM D7575 is a good stand alone method for the measurement of oil and grease as defined by the method. The method was single- and multi-lab tested following ASTM Standard Practice D2777 (Standard Practice for the Determination of Precision and Bias of

Applicable Test methods of Committee D19 on Water) and produced recoveries and precision as good as or better than EPA Method 1664A for those matrices tested and in the range of ASTM D7575 applicability (5-200 mg/L). Further, EPA is not persuaded by the technical comments it received on the method itself. For example, the representativeness of a well homogenized sample used was adequately demonstrated by the replicate studies in the validation tests. See docket number EPA-HQ-OW-2010-0192 for responses to these and all other NODA comments.

B. ASTM D7575 as an Alternative Oil and Grease Method in 40 CFR Part 136

After careful consideration of all the comments received on the NODA, EPA concludes that the case has not yet been made that ASTM D7575 should be approved for nationwide use as an alternative oil and grease method. EPA has multiple reasons supporting this conclusion. First, ASTM D7575 is not applicable to the same range and matrices as the existing Part 136 oil and grease methods. ASTM D7575 is applicable for measuring oil and grease from 5 mg/L to 200 mg/L while EPA Method 1664A is applicable for measuring oil and grease from 5 mg/L to 1,000 mg/L. Additionally, as explained in Note 2 in the method, ASTM D7575 is not appropriate for certain samples where the solid matter is not sufficiently IR transmitting, such as those that contain high levels of metal particulates. Further, EPA Method 1664A also quantifies non-polar oil and grease (SGT-HEM) while ASTM D-7575 does not.

Second and more importantly, EPA continues to share the concerns raised by many commenters. Given that the two methods measure a method-defined parameter, by definition, they cannot measure the same thing. Consequently, because of the wide variety and type of individual compounds that may be measured as oil and grease and because oil and grease is extensively incorporated in permits covering a wide variety of wastewater matrices, a permittee could be in or out of compliance simply due to a change in the test method used to evaluate

samples. EPA continues to conclude such concerns are well founded for the following reasons. First, oil and grease is a method-defined parameter. That is, the results are dependent on the particular method used. As ASTM D7575 uses a different determinative step than the existing approved methods, one would not expect the results to be the same for all applications. Second, the results of ASTM D7575 have been evaluated on a relatively limited number of matrices (13) in comparison to the extensive number and types of possible applications. In contrast, when EPA promulgated Method 1664A to replace the previous Freon-based method, it evaluated the two methods on a much more extensive and wide variety of matrices (approximately 35). Third, the data evaluated to date demonstrate that while ASTM D7575 produces results that are generally very close to the approved method for the set of matrices evaluated, they are not statistically comparable results. As such, the concerns that the two methods may produce different results are well founded.

However, EPA also recognizes that a blanket conclusion that one can never promulgate new methods for method-defined parameters based on a different determinative technique leaves little room for technology improvements. Furthermore, EPA is keenly interested in supporting the development and use of “green” methods such as ASTM D7575 that do not require solvents. As such, EPA considered various approaches for allowing its use as an alternative to approved methods while minimizing the well founded concern that the two methods may affect compliance. In other words, in those applications where the two methods produce results that are comparable enough not to affect compliance, EPA wants to encourage the use of non-solvent based methods such as ASTM D7575. On the other hand, EPA wants to prevent the use of ASTM D7575 in those applications where the two methods differ in their results and have the potential to affect a facility’s compliance status. Here, there simply is not enough data to make a

nationwide determination. Until such time that EPA has enough data to make such a determination, EPA has concluded such determinations should be made on a case by case basis rather than a nationwide basis. As a result, EPA has decided not to approve ASTM D7575 as an alternative oil and grease method in Part 136.

EPA also considered a novel approach in which it would approve ASTM D7575 as an alternative oil and grease method in Part 136 with a requirement to demonstrate comparability (side-by-side data) to the permitting authority. To determine comparability for a specific application, a permittee could use the specific side by side comparison procedures recommended in the guidance document that was developed when Method 1664A was promulgated (see "Analytical Method Guidance for EPA Method 1664A Implementation and Use (40 CFR part 136)," EPA/821-R-00-003, February 2000). Under this approach, a permittee would only be able to use ASTM D7575 if the recommended procedures demonstrated comparability. This approach would provide for a non-solvent based alternative to measuring oil and grease and eliminate the compliance concerns noted above. This approach would be novel because EPA has never approved a method for nationwide use with such a requirement. As explained in Section II.A, the purpose of promulgating Part 136 methods for nationwide use is to simplify the permitting process and reduce burden to the permittees and the permitting authority (often the state). As a result, EPA consulted with various permitting authorities on this consideration. Feedback from permitting authorities indicated that reviewing side by side comparison data would be a huge burden on the states and that many POTWs lack both the expertise and staff to conduct a side by side comparison. As a result, EPA rejected this approach.

C. ASTM D7575 as an Alternative Oil and Grease Method in Permit Specific

Applications

In EPA's effort to promote the use of newer and more efficient methods, EPA looked at a third option – the use of EPA's Alternate Test Procedures process spelled out in the regulations at 40 CFR Part 136.5. EPA considered this approach for encouraging and allowing the use of ASTM D7575 while eliminating the associated compliance concerns using existing regulatory authority. As explained in Section F, EPA recognizes that new technologies and approaches are constantly being developed and, as such, Part 136 currently allows for permittees to gain approval of the use of an alternate method for a specific application at a facility or type of discharge that is different from the approved test procedure. Therefore, the authority already exists under Part 136.5 for a permittee to request the use of ASTM D7575 as an alternative oil and grease method for a specific use (i.e. limited use ATP). The burden to review such requests rests on the EPA Regional ATP Coordinators rather than the permitting authority which is often a state or a local control authority. As such, EPA encourages permittees to carefully consider whether or not ASTM D7575 is an acceptable alternative to the existing methods for their specific matrix and, if supported by data, to make such requests to their Regional ATP Coordinator. To the extent that such requests are widespread, EPA headquarters will provide technical support to the Regional ATP Coordinators.

Part 136 already stipulates that an applicant must provide comparability data for the performance of the proposed method compared to the reference method to eliminate compliance concerns. EPA anticipates that requests for the use of ASTM D7575 as an alternative oil and grease method could be widespread, thus EPA wants to ensure that such requests are handled consistently. To that end, EPA recommends that applicants demonstrate comparability by conducting a side-by-side comparison using the specific procedures (e.g. sampling frequency, number of samples, QA/QC, and statistical analyses) recommended in the guidance document

that was developed when Method 1664A was promulgated [Analytical Method Guidance for EPA Method 1664A Implementation and Use (40 CFR part 136), EPA/821-R-00-003, February 2000]. Comparability could be shown if this side by side comparison demonstrates there is not a significant difference between the promulgated method and ASTM D7575. Finally, EPA notes that such requests may provide sufficient additional data that may allow EPA at a later date to later make a nationwide determination on the approval of ASTM D7575 as an alternative oil and grease method.

IV. New Docket Materials

1. Response to Comment document
2. Response from ASTM re: technical questions
3. Memo describing outreach to states and control authorities on burden
4. May 14, 1999 *Federal Register* (64 FR 26315)
5. “Analytical Method Guidance for EPA Method 1664A Implementation and Use (40 CFR part 136),” EPA/821-R-00-003, February 2000

Dated: February 27, 2013

Nancy K. Stoner,
Acting Assistant Administrator

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